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# Small-Scale Testing of High Bulk Cubical and Spherical Nitroguanidine for Comparative Evaluation

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**13. ABSTRACT** *(Maximum 200 words)*
Two types of nitroguanidine (NQ), high bulk cubical (HBCNQ) and spherical (SNQ), were evaluated neat and as melt cast formulations with tritonal (TNT) and with RDX/wax/aluminum. The objective was to differentiate between the two crystal habits of NQ with respect to thermal characteristics, shock sensitivity, and performance. Parameters investigated were thermal stability, critical diameter, detonation velocity and pressure, shock sensitivity, and bullet impact. Differential scanning calorimetry (DSC) thermograms suggest the HBCNQ is less stable than SNQ. This is also reflected in apparent activation energies and is supported by DSC and slow cookof data from their melt cast formulations. Shock sensitivities and detonation pressures were obtained on selected formulations. A correlation between charge diameter and that diameter, $d_1$ (NQ), above which the detonation velocity of the NQ component of the TNT-based formulation performs ideally was demonstrated. The overall results suggest that ideal performance of NQ is influenced by factors that include porosity, crystal habit, other energetic and/or nonenergetic (late reacting) ingredients, charge diameter, input pressure, and perhaps particle size and that it can be achieved more readily from a formulation containing SNQ.

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SUMMARY

This report describes the results of an investigation involving two crystalline forms of nitroguanidine (NQ) hereinafter named high bulk cubical (HBCNQ) and spherical (SNQ). The two types of NQ were tested neat and as melt cast formulations with tritonal (50 percent of each component by weight) and as a composite with RDX (21.6 percent), NQ (47 percent), aluminum (17 percent), polywax/dioctyl adipate (14.1 percent), and lecithin (0.3 percent).

The objective of the investigation was to characterize both types of NQ with respect to thermal stability, shock sensitivity, and performance. The measurable parameters chosen were thermal stability by differential scanning calorimetry (DSC) and slow cookoff characteristics, critical diameter, detonation velocity and detonation pressure, shock sensitivity (gap technique), and bullet impact. DSC thermograms obtained from neat HBCNQ and SNQ suggest the former is less stable than the latter. This conclusion is reflected in the apparent activation energies for the thermal decomposition process where that for HBCNQ is about 10 kcal/mol less than that for SNQ. This conclusion is further supported by DSC and slow cookoff experiments involving both melt cast formulations. Ideal detonation performance was shown to occur for unconfined tritonal/SNQ charges at a smaller diameter than for tritonal/HBCNQ charges. The velocity data from super large-scale gap experiments (177.8 mm ID) for those charges attaining a sustained detonation showed that both formulations (tritonal/SNQ and tritonal/HBCNQ) attained ideal performance.

The gap pressures for the two formulations suggest the one containing SNQ may be the least sensitive of the two, but neither are significantly less sensitive than tritonal alone or aluminized tritonal under these test conditions. The overall data suggest that performance of any of these formulations at any given gap pressure is influenced by factors that include porosity (charge and crystalline), crystal habit, other energetic and/or nonenergetic (late reacting) ingredients and, perhaps, particle size. It is further suggested that gap pressure is also an influencing factor with the RDX/SNQ system.

# PREFACE

This program was conducted by WL/MNME, 2306 Perimeter Road, Ste 9, Eglin AFB FL 32542-6810. The program was managed by Capt P. G. Summers and Ms. A. M. Monine, in turn, and the data analysis and interpretation performed by Dr. Robert L. McKenney, Jr. (WL/MNME). The program was conducted during the period September 1988 to December 1992.

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## SECTION I

## INTRODUCTION

### 1. BACKGROUND

Nitroguanidine (NQ) is a commercially available energetic material in the form of flexible needles ($\alpha$ polymorph), having a bulk (packing) density generally between 0.2 and 0.3 g/cm$^3$ and having a particle density of 1.64 $\pm$0.03 g/cm$^3$. This crystal habit limits its usefulness as an explosive ingredient primarily because of poor processability characteristics. The use of crystal habit modifiers and special recrystallization techniques allow the crystal habit to be changed to forms of higher bulk and particle densities and improved processability characteristics. This paper describes the results of an investigation involving two crystalline forms of NQ, hereinafter named high bulk cubical (HBCNQ) and spherical (SNQ). The former is recrystallized from water, and the latter by a special process from dimethylformamide. The sources of the two NQs are the Naval Ordnance Station, Indian Head, Maryland and Fraunhofer-Institut fur Chemische Technologie (ICT) of the Federal Republic of Germany, respectively.

HBCNQ is an irregularly shaped, polyhedral-like particle with as-received bulk and particle densities of 0.948 and 1.760 $\pm$0.008 g/cm$^3$, respectively. SNQ is an example of spherulitic crystallization with radial internal growth structure. Its bulk and particle densities are 0.976 and 1.724 $\pm$0.009 g/cm$^3$, respectively. The crystal density of NQ is 1.775 g/cm$^3$.

Both types of NQ were investigated as neat samples and as melt cast formulations with tritonal (TNT) and with RDX as the more sensitive energetic ingredients. Four particle size ranges (105-210, 210-297, 297-420, and 1-500 micrometers) were used for each type of NQ. The TNT-based formulation consisted of 50 percent by weight NQ. Homogeneous dispersion of the NQ was ensured by creaming the TNT during the formulation process. The RDX-based formulation consisted of RDX/NQ/aluminum/polywax (500)/dioctyl adipate (DOA)/lecithin (21.6/47.0/17.0/11.7/2.4/0.3 percent by weight). The Class E RDX was coated with 10 percent by weight DOA. The aluminum was atomized powder No. 1401 produced by the Aluminum Company of America, Rockdale, Texas, and the polywax was produced by Petrolite Specialty Polymers Group, Tulsa, Oklahoma.

## 2. TEST OBJECTIVE

The objective of this investigation was to differentiate both types of NQ with respect to thermal characteristics, shock sensitivity, and performance. Parameters investigated were thermal stability, critical diameter, detonation velocity and pressure, shock sensitivity, and bullet impact. This work was funded by the Foreign Technology Evaluation Program.

## 3. TEST PROCEDURES

Thermal characteristics were ascertained by differential scanning calorimetry (DSC) and slow cookoff experiments, the latter using small-scale cookoff bomb (SCB) units. The DSC experimental and data analysis techniques (References 1 and 2) and the slow cookoff unit and technique (References 1 and 3) are described elsewhere. The SCB units are lined with asphalt.

Critical diameter determinations were made using conical explosive charges as described in Reference 4 and instrumented exclusively with piezoelectric pins. Detonation velocity/pressure experiments were conducted with cast right circular cylinders of unconfined explosive. Three tests were carried out for each formulation at each specific diameter. Diameters varied from 19 to 203.2 mm for the TNT-based charges and from 25.4 to 50.8 for the RDX-based charges. Data from other selected tests, not part of this investigation, are also included. Cylinders were instrumented with piezoelectric pins for measurement of detonation velocities, while detonation pressures were obtained from the dents imparted to calibrated witness plates. Shock sensitivities were determined using the modified expanded large-scale gap test (MELSGT) and the super large-scale (SLSGT) and 8-inch gap tests in a manner similar to that described in Reference 5. The 8-inch gap test was converted to the SLSGT by removing the confinement from the donor charge and recalibrating. The densities of the Composition B donor charges varied between 1.67 and 1.70 $g/cm^3$ for all conical, bare, and confined cylindrical charges.

SECTION II

RESULTS AND DISCUSSION

## 1. THERMAL ANALYSIS

DSC thermograms associated with HBCNQ and SNQ are characterized by a broad, low intensity exotherm prior to the main exotherm and by a sharp exotherm preceded by a small endotherm, respectively. The thermogram from SNQ is similar to that from $\alpha$-NQ. In all cases, the main exotherm associated with HBCNQ occurs approximately 20°C before that of SNQ, suggesting the former is less stable than the latter. These decomposition characteristics are also reflected in the thermograms from the TNT- and RDX-based formulations and in their apparent activation energies. The similarities of the thermograms from $\alpha$-NQ and SNQ suggest the reduced thermal stability of HBCNQ may result from impurities probably incorporated during the recrystallization process.

## 2. CRITICAL DIAMETER

Critical diameter data for the four TNT-based and the RDX/HBCNQ formulations show that the TNT-based charges containing SNQ have smaller critical diameters ($19.1<d_c<25.4$ mm) than those containing HBCNQ ($25.4<d_c<50.8$ mm). No quantitative distinction can be made regarding the effect of particle size on the critical diameter, if any, for either NQ crystal habit in TNT. The slopes of the linear fits of the piezoelectric pin response data from the conical charges, when plotted versus reciprocal charge radius, all exhibit negative trends to varying degrees. The slope represents velocity decay with decreasing charge diameter and its degree, at least in part and for a given system, and is believed to suggest a diameter effect is in operation. Furthermore, the slope variation with NQ crystal habit suggests the diameter effect is primarily on the performance of the NQ component of the formulations and that the effect is more pronounced for the formulations with HBCNQ.

## 3. DETONATION VELOCITY TESTS

Average detonation velocities were obtained from the detonation velocity/pressure experiments. Charge diameters for the larger particle size SNQ and HBCNQ vary from 203.2 to 19.1 mm and for the smaller particle size material from 50.8 to 19.1 mm. Detonation velocities for the charge and for the NQ component were calculated, as was the percent of the ideal detonation velocity for NQ, $D_i$(NQ). Detonation pressures from the 2-inch diameter charges were measured. The detonation velocities for the charge and for the NQ component of the charge are calculated using the additivity principle and characteristic velocities ($D_i$); see Reference 6. The $D_i$ value for polyethylene was used for all calculations where wax was incorporated into the formulation. The calculated charge and NQ component detonation velocities for the TNT/SNQ charges of 203.2 and 177.8 mm diameter, both castings at an average 95.8 percent theoretical maximum density (TMD), clearly show that not only are these charges performing ideally but deviation from ideal performance begins at a diameter between 177.8 and 152.4 mm. The TNT-based charges containing HBCNQ, approximately 95 percent TMD, never attain ideal performance even at the largest charge diameter.

Price and Clairmont (Reference 7) demonstrated that pressed charges of pure high bulk density NQ at reduced charge densities, 91-92 percent TMD, were detonable and the NQ performed near ideally (98-99 percent $D_i$[NQ]) at diameters between 38.1 and 50.8 mm. They reported it had previously been shown that pressed charges of pure NQ at approximately 95.5 percent TMD (1.70 $g/cm^3$) could not be detonated as a 50.8 mm diameter core in a 1.27-cm-thick steel tube. It was then suggested that porosity alone could not account for this detonability phenomenon because unconfined, pressed charges of RDX/NQ (10/90) at 95 percent TMD are detonable at 50.8 mm diameter and the NQ component was stated to perform ideally. Other researchers (Reference 8) have similarly demonstrated that pressed and cast charges of TNT/SNQ (unconfined) at 95.6 through 97.5 and 94.9 through 97.4 percent TMD, respectively, were similarly detonable at a diameter of 50 mm. The performance of the SNQ, however, was less than ideal. The performance varied from 96.6 to 91 percent $D_i$(NQ), with the deviation from ideal increasing with increasing TNT content.

Additional detonation velocity data (Reference 8) generated from unconfined, cast TNT/SNQ charges of 50 mm diameter, containing varying amounts of aluminum, were reviewed and compared to calculated ideal charge detonation velocities. These data show that deviation from ideal charge velocity is increased significantly by the presence of aluminum. The overall data for unconfined charges of high bulk density NQ (pressed), RDX/NQ (pressed), TNT/SNQ/Al (cast), and TNT/SNQ or TNT/HBCNQ (cast) suggest the performance of the NQ component is influenced by a combination of factors that include porosity, charge diameter, NQ crystal habit, other energetic ingredients, other nonenergetic or late reacting ingredients, and perhaps NQ particle size.

The nonenergetic/late reacting ingredients may actually utilize some of the energy generated in the reaction zone to the extent that the NQ reaction contribution is degraded.

Limited detonation velocity data from unconfined, cast RDX/SNQ or HBCNQ/Wax/Al charges at diameters varying from 50.8 to 25.4 mm were studied. These data support the above hypothesis in that the NQ performance is significantly degraded in this RDX-based formulation containing energy-consuming wax and aluminum. The decreased performance is not only shown in the charge detonation velocities but more demonstrably in the detonation pressures for the 50.8 mm diameter charges. The crystal habit influence is more evident in this formulation, with the one containing SNQ more prone to perform ideally.

## 4. SHOCK SENSITIVITY TESTS

Test specific shock sensitivities of the TNT- and RDX-based formulations in both MELSGT and SLSGT hardware were determined. All of the observed MELSGT shock sensitivity differences are small and may be influenced by the known diameter effect. They may also reflect the lack of statistical testing and reduced quality control in charge preparation. The SLSGT charges exhibit only a 5 kbar gap pressure difference (18 versus 13 kbar) with the formulation containing HBCNQ being the more sensitive.

The comparison is to a shock sensitivity of 5 kbar for TNT and 32 kbar for AFX-1100 (TNT/OD2 wax/Al-66/16/18 percent by weight), a desensitized tritonal. Detonation velocities, from piezoelectric pin data, were used to determine run-to-detonation distances in the acceptor charges and to provide general features of the detonation reaction near the 50 percent gap distance. The latter was determined by the condition of the steel witness plate, where a hole in the plate represents a "go" condition. The reactions occurring near the 50 percent gap distance in the TNT-based formulations were characterized by very long run distances, some of which have detonation velocities barely exceeding 3 mm per microsecond for the monitored length of the charge.

Two MELSGT charges, one TNT/SNQ and the other TNT/HBCNQ, resulted in apparent low velocity detonations (LVD) of 2.71 $\pm$0.05 and 2.76 $\pm$0.08 mm per microsecond, respectively, for the monitored length of the charges. These resulted from gap pressures of 31.5 and 35.9 kbar, respectively. Only in the MELSGT experiments was there any indication of a detonation reaction beginning to fail. The reactions occurring near the 50 percent gap distance for the RDX-based formulations in MELSGT hardware are distinctly different in that the detonation reactions were noticeably failing to propagate for those charges that failed to hole the witness plate.

The detonation velocity data from the smaller diameter MELSGT charges show that only the lower density TNT-based charge with the smaller particle size SNQ and, perhaps, the RDX/SNQ charge receiving 123.3 kbar gap pressure achieved ideal detonation velocity. All other TNT-based, MELSGT charges achieved detonation velocities that corresponded to 91 to 94 percent $D_i(NQ)$ over a gap pressure range of 36 to 123 kbar. All TNT/SNQ charges in SLSGT hardware reaching sustained detonation velocity achieved ideal performance. Only a single TNT/HBCNQ charge receiving 28.0 kbar gap pressure reached ideal velocity.

The RDX-based data indicate the performance of the SNQ component is clearly influenced by the gap pressure under these test conditions, which does not appear to be the case in a pure TNT matrix. Data generated from 8-inch gap experiments with an RDX-based formulation of similar composition were consistent with this finding. The charge containing HBCNQ achieved a D(NQ) value corresponding to 70 percent $D_i(NQ)$, while the charge containing SNQ achieved ideal performance. This finding suggests that the diameter effect on D(NQ) for the RDX-based formulation containing HBCNQ is still operational at a diameter of 177.8 mm, at least within the experimental condition of 35 kbar gap pressure and 400 mm maximum charge length, and is no longer operational for the charge containing SNQ.

## 5. SLOW COOKOFF TESTS

The slow cookoff test series was internally instrumented with a type k thermocouple placed down the centerline of the explosive charge. This allowed detection of initiation of self-heating and of the temperature at which maximum reaction occurred. In general, self-heating and maximum reaction occurred at higher temperatures for all formulations containing SNQ. This is consistent with the DSC results, which suggested that SNQ and formulations containing SNQ were more thermally stable than those with HBCNQ. The violence associated with the maximum reactions was too varied to interpret.

## 6. BULLET IMPACT TESTS

The bullet impact test results are characterized by mild burns through the bullet holes for the RDX-based formulations and, generally, burns with loss of at least one end cap for the TNT-based formulations. They suggest that both formulations, regardless of the type NQ or its particle size, respond mildly to bullet impact; however, the burns from the TNT-based formulations appear to be somewhat more intense. Furthermore, the data suggest those TNT-based formulations containing HBCNQ may be more stable to bullet impact than those containing SNQ.

## SECTION III

## CONCLUSIONS

DSC thermograms showed that neat HBCNQ is less stable than SNQ, with the apparent activation energy for the thermal decomposition process being about 10,000 cal/mol less than that for SNQ. It is believed this reduced thermal stability results from impurities incorporated during the recrystallization process. These results are also reflected in the TNT- and RDX-based formulations where those containing HBCNQ exhibit lower apparent activation energies and self-heating onset temperatures. These overall data suggest NQ plays a major role in the longer duration thermal decomposition processes. Both types of formulations, regardless of NQ crystal habit or particle size, respond mildly to bullet impact, a short duration process.

The TNT-based formulations with SNQ were shown to have the smaller critical diameters ($d_c$). It was also shown that for each formulation, both TNT- and RDX-based, there was a diameter, $d_i$, at which ideal detonation velocity, $D_i$, will be achieved. $D_i$ is defined as the volume weighted summation of the characteristic velocity contributions of each of the components of the formulation, $D_i = \Sigma aD_{i(1)} + bD_{i(2)} + \ldots + zD_{i(n)}$, where a, b, and .... z are the volume fractions of the components and the terms 1, 2, .... n represent the different components. Between $d_c$ and $d_i$, a nonideal detonation with velocity D is afforded, where $D < D_i$. The overall data suggest it is the NQ component of the mixture that is performing less than ideal in this diameter range and that $D_i(NQ)$ can be achieved more readily with formulations containing SNQ.

It is believed this enhanced propensity towards ideal performance by SNQ, when subjected to a shock environment, may result, at least in part, from its greater internal porosity (inaccessible to continuous medium) per individual spherulite. This porosity may exist as rather large internal crack-like voids that appear near the center of the spherulite and/or on the surface. The more important voids, however, may be in the form of more uniform microporosity that exists within and between the individual crystallites emanating from the common center of each spherulite and, perhaps, the porosity generated on the rough surface of each spherulite due to lack of significant penetration of this surface structure by the continuous medium. This porosity, in general, may act as sites for the generation of hot spots when subjected to the instantaneous high pressure of the traveling shock wave. The heat generated by the collapse of the voids causes exothermic thermal decomposition reactions which, in turn, strengthens the shock wave, thus accelerating the overall decomposition process ultimately leading to a sustained detonation.

Ideal performance was shown to occur for unconfined TNT/SNQ charges at a smaller diameter than for TNT/HBCNQ charges. The velocity data from the SLSGT experiments (177.8 mm ID) for those charges attaining a sustained detonation showed that both formulations (TNT/SNQ and TNT/HBCNQ) attained ideal performance. The gap pressures for the two formulations suggest the one containing SNQ may be the least sensitive of the two, but neither are significantly less sensitive than TNT alone or aluminized TNT (tritonal) under these test conditions. The overall data suggest that performance of any of these formulations at any given gap pressure is influenced by factors that include porosity (charge and crystalline), crystal habit, other energetic and/or nonenergetic (late reacting) ingredients, and perhaps particle size. It is further suggested that gap pressure is also an influencing factor with the RDX/SNQ system.

## SECTION IV

## RECOMMENDATIONS

While the results from this investigation suggest differences in thermal stability and performance characteristics do exist between the two types of NQ, they are not significant. Furthermore, the shock sensitivities of the various formulations used for this investigation were not consistent with values expected from formulations deemed insensitive. Based on these Phase I results, it is recommended that this project not be extended to Phase II.

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